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SYNTHESIS AND CHARACTERIZATION OF TITANIUM METAL CARBON NANO TUBES

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ABSTRACT

Carbon Nano Tubes (CNTs) are allotropes of carbon with a nanostructure and are molecular-scale tubes of graphitic carbon with outstanding properties. Due to vast applications of CNTs, new methods and techniques are discovered to prepare carbon nano tubes and to characterize them. Certain desired properties can be inculcated in CNTs, so that they can be used in various applications. Carbon-metal Nano Tubes (CMNT) can exhibit the metallic properties to achieve high level of electrical conductivity along with the inherent properties of nano tubes. The Carbon Metal (Ti) Nano Tubes are synthesized by using egg Albumin and a Ti^{2+} salt solution. Carbon Metal (Ti) Nano Tubes so formed are characterized by UV and visible, IR and NMR spectroscopy.

KEYWORDS: Ti (II), Albumin, Albumin, Metal, Complex, IR, NMR, CNTS, CMTS NMR, AFM, STM

INTRODUCTION

The focus of our recent work is on *carbon nanotubes*; their structure, properties and uses in nano-electronic devices. Carbon nanotubes are extremely thin (their diameter is about 10,000 times smaller than a human hair), hollow cylinders made of carbon atoms. Nanotubes, depending on their structure, can be metals or semiconductors. They are also extremely strong materials and have good thermal conductivity. The above characteristics have generated strong interest in their possible use in nano-electronic and nano-mechanical devices.

Potential practical applications of CNTs such as chemical sensors, field emission, electronic devices, high sensitivity nanobalance for nanoscopic particles, nanotweezers, reinforcements in high performance composites, biomedical and chemical investigations, anode for lithium ion batteries, super capacitors and hydrogen storage have been reported.

This work is done to develop new methods and technique to prepare carbon nanotubes with desired properties. A favored method is to create Carbon-Metal Nano Tubes (CMNT) which can exhibit the metallic properties to achieve high level of electrical conductivity along with the inherent properties of nano tubes. One such metal is Titanium which can be used to create the CMNT. It is thus proposed to carry out synthesis, characterization and study of properties of Carbon Metal (Ti) Nano Tubes

However, the lack of solubility and the difficult manipulation in any solvents have imposed great limitations to the use of CNT. Indeed, as-produced CNT are insoluble in all organic solvents and aqueous solutions.

Materials and Synthesis: Stoichiometric ratios of metal and ligands are dissolved in aqueous medium and are refluxed until the complex is precipitated, and if not, the pH of the solution mixture is changed to precipitate the complex.

The Carbon Metal (Ti) Nano Tubes are synthesized by using egg Albumin and a one normal metal salt solution. The Titanium salt solution is allowed to react with amino acids present in egg albumin to form a complex of amino acids with Titanium ions. Albumin is proteinaceous in nature and is a polymer of amino acids. In amino acids, amino and carboxylic acid groups are attached on both the sides of carbon chain. Both the ends then utilized for increasing the chain length and long carbon nano tubes are formed with the complex. The metal-albumin complex then decomposed at higher temperature to give carbon metal nano tubes. The synthesized complexes were found to be insoluble in the commonly known organic solvents. Consequently, the following physical measurements and analysis were carried out to check the purity and to elucidate the structure. All the metal complexes are stable to air and moisture and decompose at very high temperatures.

PREPARATION OF AMINO ACID-METAL COMPLEX

Proteins are macromolecules comprising of amino acid as monomer. Amino acids have donor atoms on N and O present as NH2 and –COOH functional groups. Now transition elements like titanium can form complexes with donor atoms of the amino acids. These compounds on decomposition under suitable heating give carbon metal nano tubes.

Aqueous solution of Titanium salt is allowed to react with amino acid present in egg albumin the lone pair present on nitrogen of $–NH_2$ and oxygen of COO^- of COOH group present in amino acid from complex with Ti (III). In this was Titanium (III) forms cross links between two amino acid chains.

The structure of albumin is very complex and it is very difficult to produce exact structure of amino acid-metal complex. Egg albumin contain a number of different chemical compounds for example. Egg albumin contain Arginine and lysine amino acids.

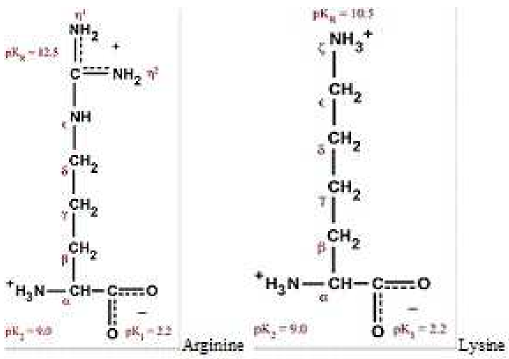
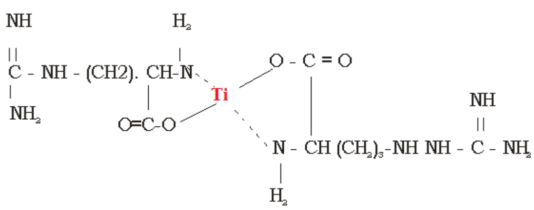


Figure 1

They react with titanium metal solution to give the following complexes.



Arginine Titanium
(III) Complex

Figure 2

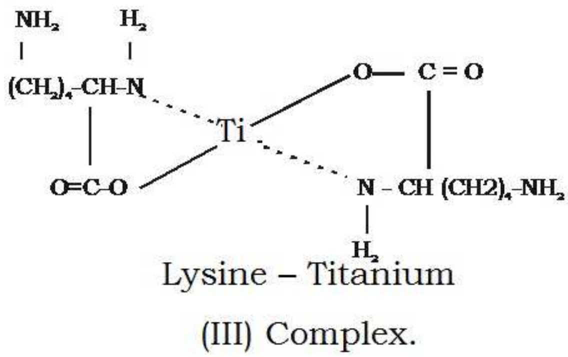


Figure 3

CHARACTERIZATION

Spectral Studies: Albumin is a complex molecule with many different molecules and hence it is very difficult to analyze the structure of this metal complex. However certain important feature can be identified which give valuable information about the structure.

IR Spectra: IR spectra of Titanium Amino acid complex is shown in the spectra 1.

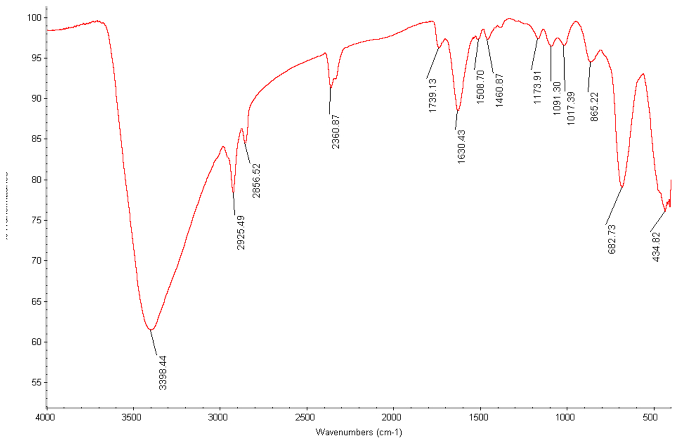


Figure 4: Spectra 1

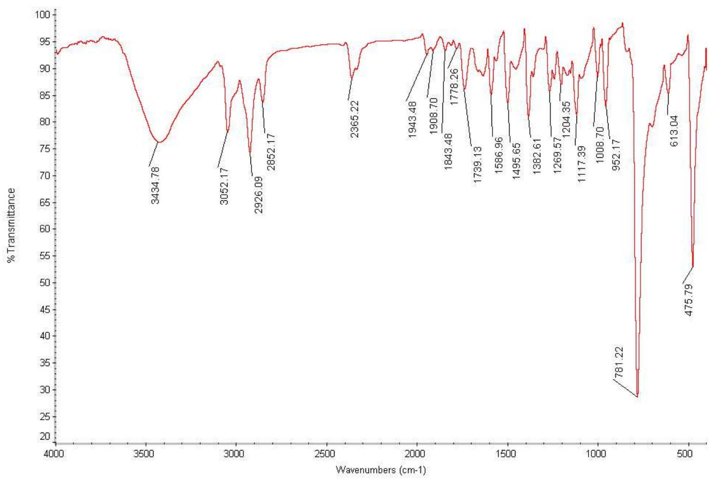


Figure 5: Spectra 2

Table 1: Typical Infra-Red Absorption Bands for Titanium Amino Acid Complex is Spectra 1

Promient Absorption Hand (CM^{-1})	Functional Group
2925.49 3398.44 1630.43 1739.13	Sp3C-H streching =N-H streching C= O streching strong signal in Amino Acid
1508.70,1460.87	NH2 or NH_3^+ asymmetric bending strong signal
1173.91	Weak C=O Symmetric streching in amino acid
1091.30	C-O streching
Spectra 2 Promient Absorption Hand (CM^{-1})	**Functional Group**
2926.09 2852.17 3434.78,3052.71 1382.61	Sp3 C-H streching Sp3 C-H streching Primary N-H streching C= O stretching strong signal in Amino Acid
1586.96,1495.65	NH2 or NH_3^+ asymmetric bending strong signal
1117.39	Weak C=O Symmetric stretching in amino acid
1117.39	C-O streching

In infrared spectroscopy, a weak band around the 3400 Cm^{-1} assigned to the N-H streching vibration. C=O streching strong signal in amino acid occurs at the 1730-1755 Cm^{-1}. The strong intense bands 1600Cm^{-1} of metal amino acid complex can be attributed to NH_2 or NH_3^+ asymmetric bending and also a strong intense band 1481-1550 Cm^{-1} is assigned to the NH_2 or NH_3^+ symmetric bending. The bands at 1400 cm^{-1} of metal amino acid complex indicates the presence of weak C=O symmetric stretching in amino acid. The band at 1100Cm^{-1} can be assigned to the C-O stretching in titanium amino acid complex.

NMR Spectra

NMR Spectra of Titanium Amino acid complex is shown in spectra 2. The information which we get by the NMR Spectra is summed up in the table 2

Table 2: Characteristics Proton Chemical Shift Spectra 1

Types of Proton	Chemical Shift S(PPM)
R. CH_2	1.289
H-C-C=O	2.346
NH_2	3.207
NH2- Ti	4.848

Characteristics proton chemical shift

Table 3: Spectra 2

Types of Proton	Chemical Shift S(PPM)
R. CH_2	1.42
H-C-C=O	2.416
NH_2	3.306
=NH	5.344

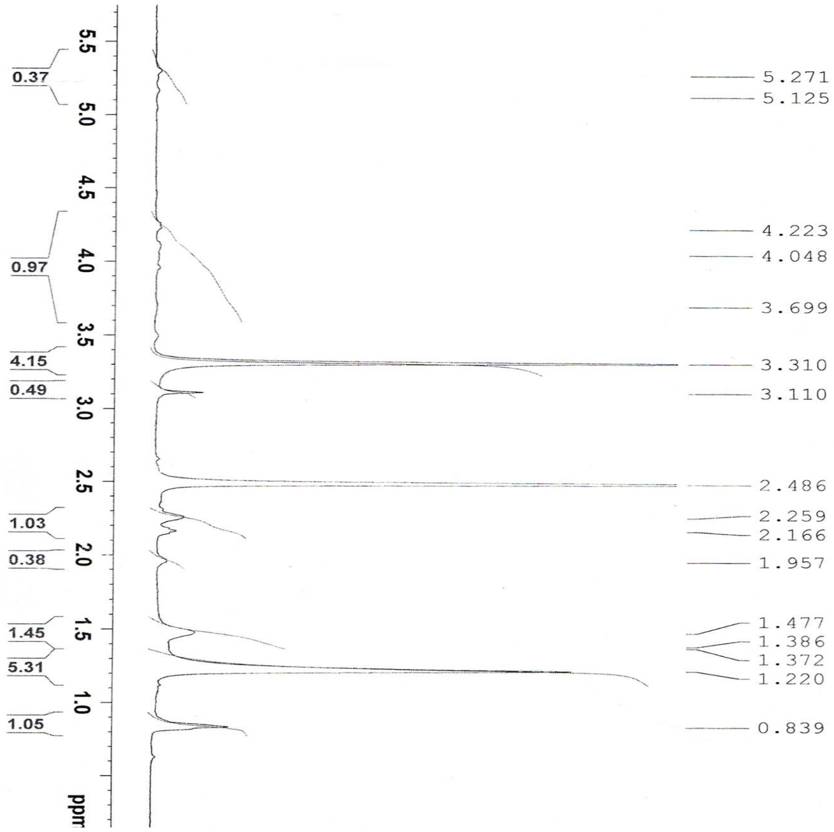


Figure 6

Figure 7

In the [1]HNMR spectra of the synthesized titanium Amino acid Complex with chemical shifts within the range 3.3 pm the NH_2 Protons was observed. In the spectra, signal with chemical shift at 2.416ppm C-H Protons attached to the COOH in the amino acids was observed. In the spectra signal with chemical shifts at 1.42ppm1.42ppm is related to the R CH_2 (secondary type) Protons and the signal with chemical shift with the 0.839 ppm corresponds to the R.CH_3 (Primary type) protons also observed.

Characterization by Scanning Probe Instrument

AFM (Atomic Forces Microscopy)

Atomic force microscopy (AFM) or scanning force microscopy (SFM) used to measure the size of the particles. It has very high-resolution type of scanning probe microscopy, with demonstrated resolution on the order of fractions of a nanometer, more than 1000 times better than the optical diffraction limit. The length of the nanotube obtained by break down of the Ti metal complex is measured near about 10^{-9} nano dimension.

STM (Scanning Tunneling Microscopy)

The scanning tunneling microscope (STM) is a type of electron microscope that shows three-dimensional images of a sample. It measures the amount of electro current flowing between a scanning tip and a surface. By the STM measurement, electrical conductivity characteristics of prepared Titanium metal-carbon nano tubes are determined.

RESULTS AND DISCUSSIONS

Carbon Titanium metal nano tubes are synthesized by the decomposition of amino acid- titanium complexes. These carbon metal-metal nano tubes are formed possess electrical conductivity due to the presence of unpaired electron. IR, NMR spectra confirms the structure of the amino complex.

CONCLUSIONS

The metal carbon nanotubes can be synthesized by chemical method. The characteristics and structure of the CNT's formed are determined by using NMR, IR spectra, STM and AFM scanning.

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